

THE MICROSTRUCTURE AND CHARACTERIZATION OF TiN COATINGS

PREPARED BY PHYSICAL VAPOUR DEPOSITION (PVD)

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ABSTRACT

Titanium nitride (TiN) possesses excellent property as corrosion resistance, aesthetic appearance and so on. Titanium nitride (TiN) is being used for coating materials on SS, Mild steel. Since many techniques are there amongst, reactive DC magnetron sputtering has many advantages to deposit ceramic coatings using metallic targets.

In the present work, our main aim is to improve the hardness of mild steel. The titanium of 2" diameter and 3 mm thickness is used as a target (99.99 % purity) for the deposition of TiN coatings. The micro structural, mechanical and wear properties are investigated using XRD, SEM, Vicker hardness tester and pin on disc respectively.

KEYWORDS: TiN Coatings, Microstructure & Corrosion Properties

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1. INTRODUCTION

Table 1.1: Properties and Structure of Tin

| | |
|-------------------------------|--------------------------------------|
| Chemical formula | TiN |
| Crystal structure | Cubic |
| Thermal expansion coefficient | $9.35 \times 10^{-6} \text{ K}^{-1}$ |
| Melting point | 2,930 °C |
| Hardness | 1800-2100 HV |

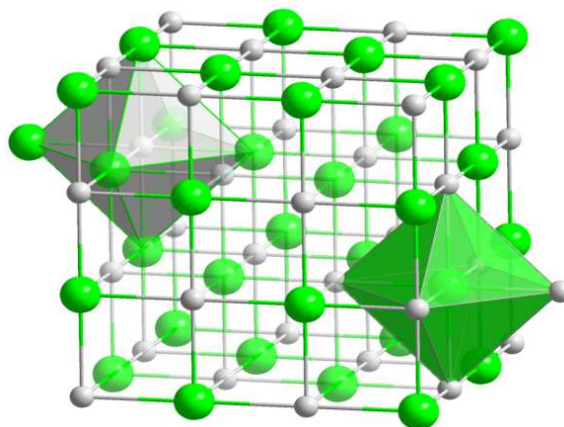


Figure 1.1: FCC Structure of the Tin

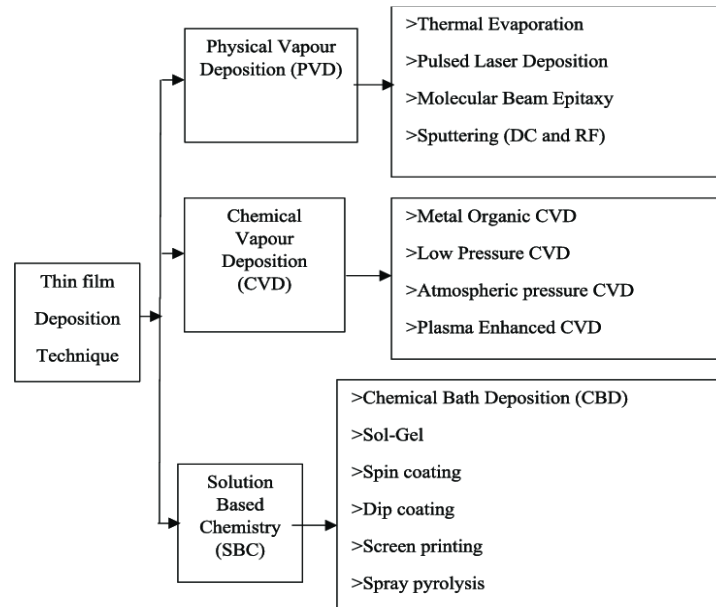


Figure 1.2: Classification of the Most Common Deposition Techniques

2. EXPERIMENTAL DETAILS

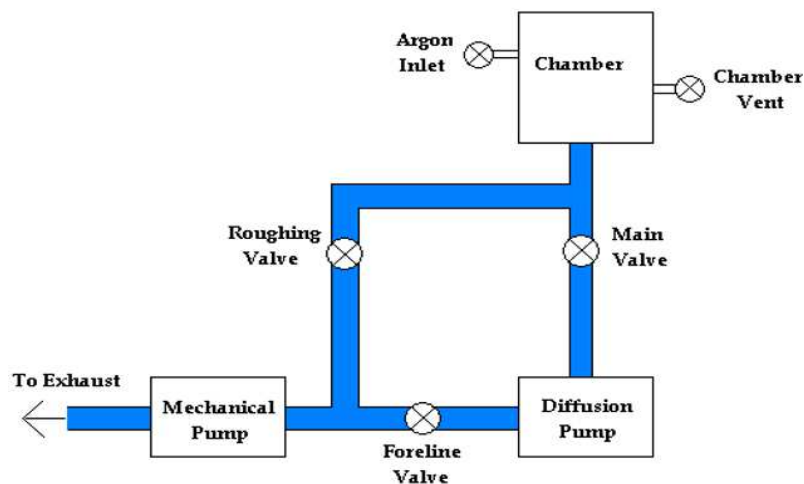


Figure 2.1: Schematic Diagram of the Sputtering Unit

3. SPUTTERING PROCESS

In this case of sputter, the deposition is we eject a material from a stipulated point, a source and later on added on to such as a metal substrate

Sputtering Process

During sputtering process the Sputtering starts when a negative charge is applied which then on to the target material causing a plasma or glow discharge.

Hence the Positive charged gas ions are generated in the plasma region and gets attracted to the negatively biased target plate. Here the collision creating a momentum transfer and later ejects atomic size particles form the target.

So on these particles traverse the vacuum chamber and are deposited as a thin film on the surface of the substrates.

As more and more atoms coalesce on the substrate, they begin to bind to each other at the molecular level, forming a tightly bound atomic layer. Here we allow more than one layer which is created for the actual production of thin film structures.

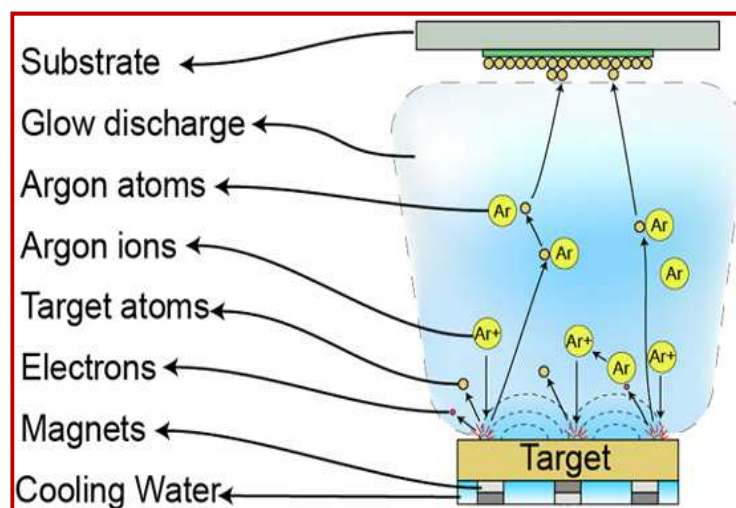


Figure 2.2: Principle of Sputtering

3.1 Deposition Parameters

- DC Power
- Argon gas pressure
- Target-substrate spacing
- Ambient gas/pressure
- Substrate temperature

3.2 Deposition Conditions

| | |
|------------------------------|-----------------------------|
| DC Power | : 400V, 0.2 Amp |
| Argon flow rate | : 30 sccm |
| Nitrogen flow rate | : 10 sccm |
| Substrates | : MS |
| Substrate temperature | : RT, 250 & 500°C |
| Deposition time | : 1 hour |
| Target | : Titanium |
| Base vacuum | : 3.0×10^{-6} mbar |
| Deposition pressure | : 5.0×10^{-3} mbar |
| Substrate to target distance | : 80 mm |

3.3 Experimental Procedure of Wear Test

Here in this experimental procedure of wear test pin being held on a disc.

The following parameters are load, speed and distance are the three parameters, such that basically the pin and then the disc are cleansed thoroughly before the actual starting of the experiments.



Figure 3.1: Pin in Disc Wear Test Rig

4 RESULTS AND DISCUSSIONS

4.1 Thickness Measurement: Dektak Profilometer

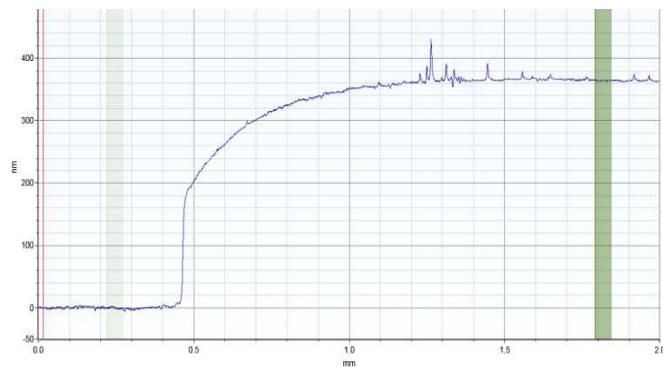


Figure 4.1: Thickness of the Tin Coating

Dektak profilometer is capable of measuring step heights between 100 Å and ~50 microns with a resolution of 10 Å [43]. The thickness of the TiN coatings on silicon is measured by using the Stylus profilometer and is found to be in the range 300-350 nm.

4.2 X-Ray Diffraction (XRD)

The angles 45.26°, 46.15°, 52.3°, 42 and 76.0°, corresponding to (111), (200) and (220) reflections indicated formation of polycrystalline tetragonal structure of TiN thin films. X-ray diffraction datas tells sample containing Titanium nitride in face centered cubic structure (PDF 38-1420). Here the intensity of the peaks denotes the increased crystallinity.

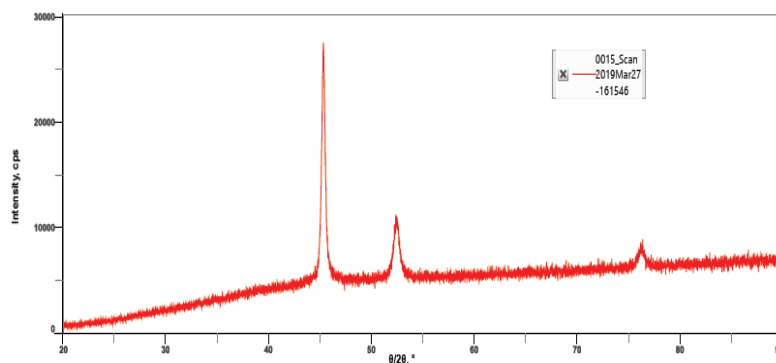


Figure 4.2: XRD – TIN Film-500°C

4.3 SEM

Scanning electron microscopy is used to characterize surface topography of the sample. The morphological study is very important for the films to observe the changes in the microstructure. Figure show the SEM image, indicating the surface topography of the TiN nanostructured thin films.

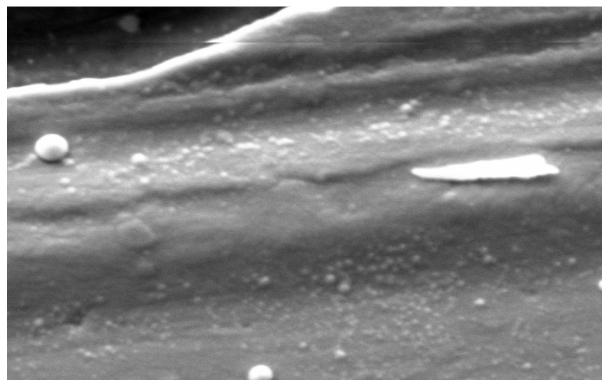


Figure 4.3: SEM Image



Figure 4.4: Wear Test of the Tin Film Deposited at 500°C Temperature

5. CONCLUSIONS

The Titanium nitride films which are deposited at different temperatures were investigated in this work. The XRD, SEM, micro indentation and pin on disc methods were employed to characterize the micro structural and mechanical properties of the films. Results shows the mechanical and wear properties of TiN films. XRD studies indicated the formation of polycrystalline TiN with fcc structure. The SEM studies show the formation of the crystallites with uniform distribution. The increase in the hardness values of TiN films with the increase of temperature was attributed to the dense and fine grain structure. The COF is reduced at the higher deposition temperature.

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